In the Application of: Dr. Oskar K. Wack et al

Ser. No.: 09/142,452

Filed: January 19, 1999

For: METHOD OF CLEANING OBJECTS

Examiner: Alexander Markoff

Group: 1746

Assistant Commissioner for Patents
Washington, DC 20231

DECLARATION UNDER 37 C.F.R. 1.131

Dear Sir:

We, Dr. Oskar K. Wack, Mr. Martin Hanek, and Mr. Karsten Lecamann, declare as follows:

1. We are the inventors of the subject matter described and claimed in the above-identified US Patent Application Serial No. 09/142,452. We can read and understand English and have read and understood this declaration.
2. The acts relied upon to establish our earlier date of invention were carried out in a WTO member country, namely Germany.
3. Attached hereto as Exhibit A is a true and accurate copy of pages from the laboratory notebook of Hildegard Nagy, a chemical engineer employed by the assignee company, who performed the following experiments according to our instructions and under our supervision. Because the notations were made in German, English translations are provided as appropriate.
4. Prior to September 19, 1998, we actually reduced to practice the invention as presently claimed in the above-identified application. For example, prior to September 19, 1998, a solution of 10% dipropylene glycol mono n-propyl ether ("DPnP") in water was prepared according to our instructions and physical

properties of this solution were observed. For example, see page 1 of the translation of the lab note book. In particular, we observed that this solution was clear at room temperature and was a cloudy two-phase solution at about 30°C.

5. A solution of 10% Dowanol DPnP in H_2O was also prepared and physical data were determined (see page 2 of the translation of the lab note book).

6. The cleaning ability of these solutions was evaluated by applying ultrasound to the solutions in order to clean flux from soldered circuit boards (see page 2 of the translation of the lab note book). The application of ultrasound served to agitate the solution and maintain the solution in the state of organic-rich droplets dispersed within a continuous aqueous phase. As a result of these tests, we determined that DPnP and similar compounds mixed in water were excellent cleaning solutions that effectively cleaned both hydrophobic and hydrophilic contaminants from objects. As noted above, all these acts occurred in Germany before September 19, 1998.

7. We further declare that all statements herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above-referenced application or any patent issuing thereon.

DATE: 14 May 2003 By: _____
Dr. Oskar K. Wack

DATE: 14 May 2003 By: _____
Mr. Martin Hanak

DATE: 14 May 2003 By: _____
Mr. Karsten Zessmann

7.04.96.

Versuchsdurchführung: Metallstäbe mit Gummiöl werden in das obige Gemisch bei ca. 80°C mit US gerührt, gespült danach mit Bauseren Gemisch bei 85-90°C mit US

Ergebnis - starker Angriff auf Metallstäben.
Korrosionsrückstände mit Metallstäben.

- Purasalo ML 25% + 10% Dow DPnP in H_2O + 0,1% Chromat (bei 90°C)
- Ergebnis - leichter Angriff.

- Purocler ML 25% + 10% DPnP + 2,5% Butoxal in H_2O (bei 90°C)
Ergebnis - kein Angriff, jedoch Produktrückstände.

13.02.96.

- Purasalo ML 25% + 10% DPnP in H_2O (bei 50°C)
+ kein Angriff, jedoch Flecken.

- Purasalo IPL 36% + 10% DPnP in H_2O (bei 50°C)
- kein Angriff, keine Flecken.

- Purasalo IPL 35% + 10% DPnP in H_2O (bei 90°C)
- Korrosion.

16.02.96

Dow DPnP 10% in H_2O bei RT - klar. bei etwa 30°C - trüb 2 Phasen
Durch Zugabe von 5% Dow DPnP + wiederholter Aufheizung
Produkt klar, durch erneutes Aufheizen wird Produkt wieder trüb.

Dow DPnP 10% in H_2O - Flammpunktbestimmung erfolgte ohne Rühren zu bestätigen → nicht bestimmbar.

3% Dow DPnP in H_2O - beim Erhitzen (bei 40°C trüb) - schwimmen Fettaugen oben auf.
8% Dow DPnP in H_2O - durch Erhitzen wird Produkt trüb (65°C)
- Fettaugen auf der Oberfläche.
7% Dow DPnP in H_2O - bei 50°C trüb → weniger Fettaugen als bei 8%.

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Signed Date Signed Date

16.02.96

[redacted]

Exhibit A - S/N 09/142,452 - attached to declaration under 37 C.F.R. 1.131 signed May 14, 2003
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PROJECT DR. O. K. WACK-CHEMIE

Notebook No. Nr. 8257 S. 4/4 43

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6% Dow. DPnP in H_2O – bei 56°C – trüb
5% Dow. DPnP in H_2O – bei 65°C – trüb

21.02.96

10% Dowanol DPnP in H_2O ≙ Zestron VD 200 Rez Nr 1.

Physikalische Daten:

Dow DPnP – 10
VE-H_2O – 90

- Dichte – 0.997 g/cm³
- Oberfläche – 34.6 mN/m
- Visko – 16 cSt.
- Brechind. 13474
- Flammpunkt – nicht bestimmbar
- Gefrierpunkt = – 2°C.
- Siedepunkt = 100°C

26.02.96

11.03.96

Überprüfung der Reinigungswirkung von Zestron VD 200 + 0.3% A-butanol im Vergleich zu Zestron VD 200 neue Zestron FA.

Testobjekt – Platinen mit unterschiedlichen ... Flussm. gefluxt.

Reinigungsdauer : 3 min US bei 70°C bzw 50°C für Zestron FA
Spülung : 3 min. US mit Zestron VD 200 bzw H_2O im Reinigungsprozeß mit Zestron FA

Bewertung:
bei der Reinigung mit Zestron VD 200 mit den Flussmittel
1F 2005 –
CCP L3 – } Ablagerungen trüb. Rückstände.
α-grillo –

Zestron VD 200 mit 0.3% A-butanol:
1F 2005 –
CCP L3 – } keine Verbesserung zum Standard.
α-grillo –

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12.11.95.

Mischung: Wasser / Lösungsmittel

Dowanol PM 53 % in H_2O - F_p = 53°C.

11.12.95.

Dowanol DPM 8,9 % in H_2O F_p = > 100°C

04.12.95 Glt

06.12.95.

Cyclopentanon 57,6 in H_2O F_p - 34°C

Furfurylalkohol 20 % in H_2O F_p > 100°C.

Flammpunkt nicht bestimmbar

11.12.95

Propoxypropanol (Dow PNP) 42 % in H_2O F_p - 49°C.

11.01.96.

Furfurylalkohol 20 % + 5 % Dimethylacetal in H_2O F_p - nicht bestimmbar.

15.01.96 Glt

23.01.96.

Tetrahydrofurfuryl 10,5 % H_2O F_p - nicht bestimmbar.

Dow DPnP 9,6 % in H_2O F_p - nicht bestimmbar.

Furasalv HL 20 % H_2O F_p - nicht bestimmbar

19.01. 22.01.96.

Reinigungsversuche mit Reaktor Mischung / Furfurylalkohol 20 % in H_2O.

Ergebnisse nicht auswertbar.

8.02.96.

Furasalv HL 25 % + Dow DPnP 10 % in H_2O - nicht bestimmbar.

Reinigungsversuche mit Reaktor Mischungen wie:

HL 25 % + Dow DPnP 10 % in H_2O

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14.10.96 Glt

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[redacted]

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Official translation MPC r lated -Lab notes

9.02.96 (= Feb. 9th 1996)

Setup for the conducted experiment: Metal bars contaminated with oil residues.
Are cleaned in the azeotrope mixture with ultrasound(US) at 85°C
Rinsed with clean mix (refers to azeotrope mix) at 85-90°C with ultrasound.
Result: Strong surface attack (surface penetration) on metal bars.

Corrosion related experiments with metal bars:

- Purasolv ML 25%with 10% DPnP in H_2O plus 0.1 Chromate (at 90°C)
Result: slight metal attack

- Purasolv ML 25%with 10% DPnP and 2.5 Aminobutanol in H_2O (=water) (at 90°C)
Result: no attack visible, however product residues visible.

13.02.96 (= Feb.13th 1996)

-Purasolv ML 25% - 10% DPnP in H_2O (at 50°C)
Result: no attack, but spotty residues

-Purasolv IPL 35% - 10% DPnP in H_2O (at 50°C)
Result: no attack, no spots or residues

- Purasolv IPL 35% plus 10% DPnP in H_2O (at 90°C)
Result: Corrosion.

16.02.96 (= Feb. 16th 1996)

Dow DPnP 10% in H_2O at RT – clear. At around 30°C – cloudy and two separate phases – Addition of 5% DPNP product clear, ~~repeated heating cycles~~ through renewed heating the product turns cloudy again.

Dow DPnP 10% in H_2O – Determination of the flashpoint was done without agitation of the stirrbar → unable to determine flashpoint.

9% DPnP in H_2O – through heating (turns cloudy at 42°C) – oily/grease like-droplets are visible on the surface.
8% DPnP in H_2O – with heating the product turns cloudy (45°C). oily/grease-like droplets on the surface.
7% DPnP in H_2O – turns cloudy at 50°C → fewer oily/grease like-droplets as with 8% scenario.

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6% Dow's DPnP in H_2O – cloudy at 56°C - ...
5% Dow's DPnP in H_2O – cloudy at 63°C -...

21.02.96 (= Feb.21st 1996)

10% Dowanol DPnP in H_2O, which will be named Zestron VD200- recipe Number1

Physical Data:
Density – 0.997 g/cm3
Surface tension – 34.6 mN/m
Viscosity – 1.6 cSt
Refractive Index – 13474
Flashpoint – cannot be determined
Freezing point = - 2°C
Boiling point = 100°C

26.02.96 (= Feb. 26th 1996)

11.03.96 (= March 11th 1996)

Evaluation of the cleaning ability of Zestron VD200 plus 0.3% amino-butanol in comparison to Zestron VD200 as well as Zestron FA.

Test object – soldered circuit boards with different low solid content fluxes.
Cleaning time: 3 minutes with ultrasound at 70°C and at 50°C for Zestron FA respectively.
Rinsing: 3 minutes ultrasound with Zestron VD200, DI water respectively. In the wash tank with Zestron FA

Results obtained:

Cleaning with Zestron VD200 with the following fluxes:
IF2005 –
CCP L3 – => Solder pads/leads dulled, residues visible
α-grillo –

Cleaning with Zestron VD with 0.3% A-butanol (=aminobutanol)

IF2005 –
CCP L3 – => no improvement visible when compared to the standard.
α-grillo –

18.03.96 (= March 18th 1996)

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17.11.95 (= October 17th 1995)

Mixture: Water/solvent

Dowanol PM 53% in H_2O - Flashpoint = 53°C

Dowanol DPM 8,9% in H_2O - Flashpoint = >100°C

04.12.95 (= Dezember 4th 1995)

Cyclopentanon 57,6% in H_2O Flashpoint 34°C

Furfurylalcohol 20% in H_2O Flashpoint > 100°C
Flashpoint could not be determined.

11.12.95 (= Dezember 11th 1995)

Propoxypropanol (Dow PNP) 42% in H_2O Flashpoint 49°C

11.01.96 (= January 11th 1996)

Furfurylalcohol 20% in H_2O plus 5% Amino butanol in H_2O Flashpoint could not be determined

15.01.96 (= January 15th 1996)

Tetrahydrofufurylalcohol – 10.5% in H_2O		Flashpoint could not be determined
Dow DPnP	- 9.6% in H_2O	Flashpoint could not be determined
Purasolv ML	- 20% in H_2O	Flashpoint could not be determined

19.01.96 (= January 19th 1996) **22.01.96** (= January 22nd 1996)

Cleaning trials with Azeoptrope mixture of Tetrahydrofufuryl alcohol 20% in H_2O
Results not satisfactory.

8.02.96 (= February 8th 1996)

Purasolv ML 25% with Dow DPnP 10% in H_2O – not able to determine results
Cleaning trials with azeotropic mixtures as for example:
ML 25% with Dow DPnP 10% in H_2O.

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I, Sylvain Chamousset, certify under penalty of the laws of the United States, that I am competent to translate from the German language to the English language and that the above is a true and correct translation into English of the German language document " Labor Versuchsdurchfuehrungen " attached hereto.

Mr. Sylvain Chamousset

March 27, 2003

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